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THE EFFECT OF POTASSIUM CHROMATE ON THE ANAEROBIC DIGESTION OF SEWAGE SLUDGE

A THESIS

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Master of Public Health Engineering

By

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ON THE
ANAEROBIC DIGESTION OF SEWAGE SLUDGE

APPROVED:

Date Approved by Chairman: 7 September 1956

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SUMMARY

It is generally known that chromate is toxic to microorganisms. Because chromates are discharged from industries to sewers, the extent of their toxicity to the anaerobic digestion of sewage sludge has been studied. However, a great variety of results has been reported from previous investigations. Whereas one investigator claims that 200 p.p.m. of precipitated chromium in sludge will noticeably reduce the rate of digestion, other investigators have shown that 10,000 p.p.m. hexavalent chromium may be tolerated by the digesting sludge. Concentrations intermediate to these have been held to produce toxicity in various degrees. Because chromate ions are reduced to chromic ions during digestion, both forms of chromium were considered in this study.

The objectives of the present study were designed both to explain the wide divergence in toxicity obtained previously and to understand the mechanism of the toxic action. Organic matter is converted to methane and carbon dioxide during anaerobic digestion, and the efficiency of sludge digestion was determined in this investigation by the measurement of daily gas production.

The results obtained indicate that the biological condition of the sludge is of primary importance in determining the extent of the toxicity of chromate on the digestion process; consequently the disagreement in toxicity reported in the literature may be explained by the fact that the biological condition of the sludge was different in each study. In this study, by increasing the temperature of digestion

or by allowing the organisms of digestion to flourish before adding the chromate, the activity of digestion was increased with the result that the toxicity of chromate was decreased.

Chromate ions prevented the production of gas, whereas the chromic ions produced during digestion were much less toxic. Recovery of the activity of the digestion, either partial or complete, was effected only after the chromate present was reduced. A sludge with a low concentration of organic matter underwent a poorer digestion than a more concentrated sludge, each sludge containing equal concentrations of chromium. From an analysis of relative toxicities disclosed, it is suggested that 200 p.p.m. chromium should be the maximum concentration allowed in an anaerobic digestion tank in one infrequent dose.

Further study is recommended on the factors of pH, organic content, gas analysis, ratio of digested sludge to raw sludge and temperature to determine more fully their relation to chromate toxicity in sludge digestion.

CHAPTER I

INTRODUCTION

Sludge digestion is the process in one phase of sewage treatment whereby unpleasant and potentially infectious sewage sludge is rendered practically inoccuous and readily disposable. The sludge, which settles from the sewage in a quiescent tank, is digested in the absence of air by microogranisms which may be already included or are seeded into the sludge.

In most practices the procedure is to add a small amount of raw sludge to a larger amount of well-digested sludge which furnishes great numbers of the essential methane organisms and, in addition, provides a suitable environment with regard to pH and oxidation-reduction potential. During the digestion large amounts of organic material are converted into methane and carbon dioxide, the measurement of which is used as the principal indicator of the progress and efficiency of digestion. In sewage plant practice the gaseous mixture may be used as a fuel for heating the digestion tanks or for power generation.

Anaerobic digestion consists of two distinct phases. One phase is the production of volatile acids from the breaking down of such substances as sugars, starches, cellulose, and nitrogenous compounds by organisms which are common to sewage sludge. This phase is accompanied by the production of small quantities of gas, chiefly carbon dioxide. The other phase is the utilization of these acids, and certain accompanying by-products, by the methane organisms to produce methane and

carbon dioxide. The two phases occur separately in unseeded digestion and simultaneously in well-seeded digestion.

A pH below about 6.5 will retard gas production, and an important role of the digested sludge is to maintain neutrality by furnishing sufficient quantities of buffer (ammonium bicarbonate, principally). Also, the methane fermentation proceeds at a low oxidation-reduction potential, and digested sludge contains compounds, such as hydrogen sulfide, which exert a poising effect in maintaining the low oxidation-reduction potential. The presence of strong oxidizing agents, such as nitrates or oxygen, in the raw sludge will interfere with the digestion process. In order that the methane organisms may function efficiently, the ratio of digested sludge to raw sludge must be controlled such that the pH remains near neutral and the oxidation-reduction potential remains low.

The rate of digestion depends to a great degree on the temperature of digestion. Digestion may be carried out in two different ranges of temperature, the fermentation in each range being performed by different types of organisms. Mesophillic digestion has an optimum temperature of about 100°F., while thermophillic digestion has an optimum temperature of about 130°F. (1). Nevertheless, most heated digestion tanks are operated at temperatures between 85° and 95°F. because of operating difficulties encountered at the higher temperatures.

The methane organisms are sensitive to other changes in their environment, and many undesirable materials will interfere with or terminate the digestion. Various inorganic salts are among these interfering substances (2). The term toxicity as used in this thesis

will mean the degree of interference with the digestion process by specific agents.

It is generally known that chromates are toxic to the organisms of digestion. In addition to the effect of chromium as a heavy-metal cation, the factors of oxidation-reduction potential of the chromate anion and the pH effects of each form are involved in this study. The oxidizing nature of chromate is such that the oxidation-reduction potential of the fermentation is disturbed. The oxygen of the anion may contribute to the destruction of organic matter without the production of the normal anaerobic by-products (organic acids). When potassium chromate is dissolved in water, the resulting pH will be in the mildly alkaline range. The pH of a 10 per cent solution will be about 8.5. During early stages of a batch digestion to which chromate has been added, the chromate ions are reduced to chromic ions and the pH falls. The pH situation is highly involved because of several factors. Potassium hydroxide, remaining when the potassium chromate is reduced, is stable and tends to keep the pH high. Chromic ions combine with hydroxyl ions to form insoluble chromic hydroxide, thus removing hydroxyl ions from the solution and tending to lower the pH. Even if chromic ions are held in solution by means of chelation, the chelating agents are subject to digestion and may be destroyed. The chromate ions are toxic to the methane fermentation phase; this fact causes the production and accumulation of organic acids with a lowering of the pH.

Large quantities of chromates are discharged daily from such industries as plating shops and tanneries, and many plants discharge their chromate wastes directly into municipal sewers. In fact, Pettet (3) has asserted, "Wherever possible, discharge of plating wastes into a sewer,

with or without pretreatment as required, is the safest method of disposal." A considerable portion of these chromate wastes unavoidably finds its way into the sludge digestion tanks. Because these discharges are associated with poor sludge digestion, several investigations have been made to determine the extent of toxicity of chromates on the digestion process. Investigations of the effect of chromic ions on sludge digestion are also of interest in this discussion, because chromate ions are reduced to chromic ions during the digestion.

A series of laboratory experiments performed at Wesleyan University have shown (4,5) that 200 p.p.m. of precipitated chromium in sludge will noticeably reduce rate of digestion. On the other hand, Barnes and Braidech (6) have reported that 2,000 p.p.m. of trivalent chromium retarded digestion only 11.6 per cent, while 5,000 p.p.m. of hexavalent chromium retarded digestion only 41.0 per cent and 10,000 p.p.m. hexavalent chromium retarded digestion by 68.4 per cent. A Dutch investigator, Vryburg (7), has observed that 500 p.p.m. chromium (as chromium sulfate) reduced gas production by one-half, while 1,000 p.p.m. reduced the normal gas production by five-sixths. It should be noted that sulfate ions are themselves toxic to the digestion process in higher concentrations by contributing oxygen and toxic sulfide (8).

Several investigators have assigned values to the maximum concentration of chromium permissible in the total sewage. This value must be considered, because it has been observed (9) that reduced chromium compounds accumulate in the sludge. Such varying values as 1 p.p.m. (4,5), 5 p.p.m. (10,11) for total chromium and 26 p.p.m. (12) for chromate have been reported as being limiting concentrations.

Graham (13) has stated that chromic salts are much less toxic than chromates and should have no effect on sewage treatment processes in low concentrations. Coburn (9) has stated that chromium in the chromic state will precipitate almost quantitatively in sewage and will be removed to the digestion tanks with the sludge, where the digestion will be retarded.

Hoover (4) has observed that soluble chromates show much less interference with sludge digestion than precipitated chromium, although the persistent yellow color of chromate is objectionable. Coburn (9) has also reported that chromates cannot be precipitated under ordinary conditions and, therefore, this form of chromium will effect sludge digestion only slightly. He infers, however, that, if present, chromates would be very toxic to anaerobic digestion.

In a study of the effect of chromium on the methane fermentation of acetic acid, Pagano, Teweles and Buswell (14) found that hexavalent chromium exerted a greater toxicity than trivalent chromium and that both forms of chromium were precipitated. He disclosed that daily dosages of 1.5 p.p.m. hexavalent chromium caused decreases in the activity of the fermentation after three to five additions and that rates of fermentation returned to normal after a period of recovery. He further discovered that an initial dose of from 50 to 200 p.p.m. trivalent chromium could not be followed by additional daily dosages without first allowing sufficient time for recovery.

The objectives of the present study were designed both to explain the wide divergence in toxicity previously reported and to underatand the mechanism of the toxic action. A comprehension of the factors in-

volved is necessary for the intelligent control of the quantity of chromate which may be allowed to enter sludge digestion tanks.

CHAPTER II

INSTRUMENTATION AND EQUIPMENT

The efficiency of sludge digestion was determined in this investigation by the measurement of daily gas production of samples with and without chromate. The gas was collected and measured by means of displacing water from a calibrated tube. The toxicity was evaluated by observing the difference in gas production between the samples with and without chromium.

The apparatus used for an individual sample is diagramed in Figure 1. A two-liter glass bottle contained approximately one liter of a mixture of raw and digested sludge. The gas produced by the digesting sludge passed through the connecting tubing to a one-liter glass tube which was calibrated at intervals of 25 milliliters, each reading being estimated to the nearest five milliliters.

Before each run, the calibrated tube was filled with water and sealed at the top by a clamp. The water remained in the tube until the gas of fermentation displaced it. Since the atmospheric pressure was much greater than the hydrostatic pressure developed by the water in the tube (about three feet of water), two methods of keeping the water in the tube were considered; in one technique the tube could have opened beneath the surface of a reservoir open to the atmosphere, or the opening at the bottom of the tube could have been of such a small size that the surface tension would have prevented the escape of the water.

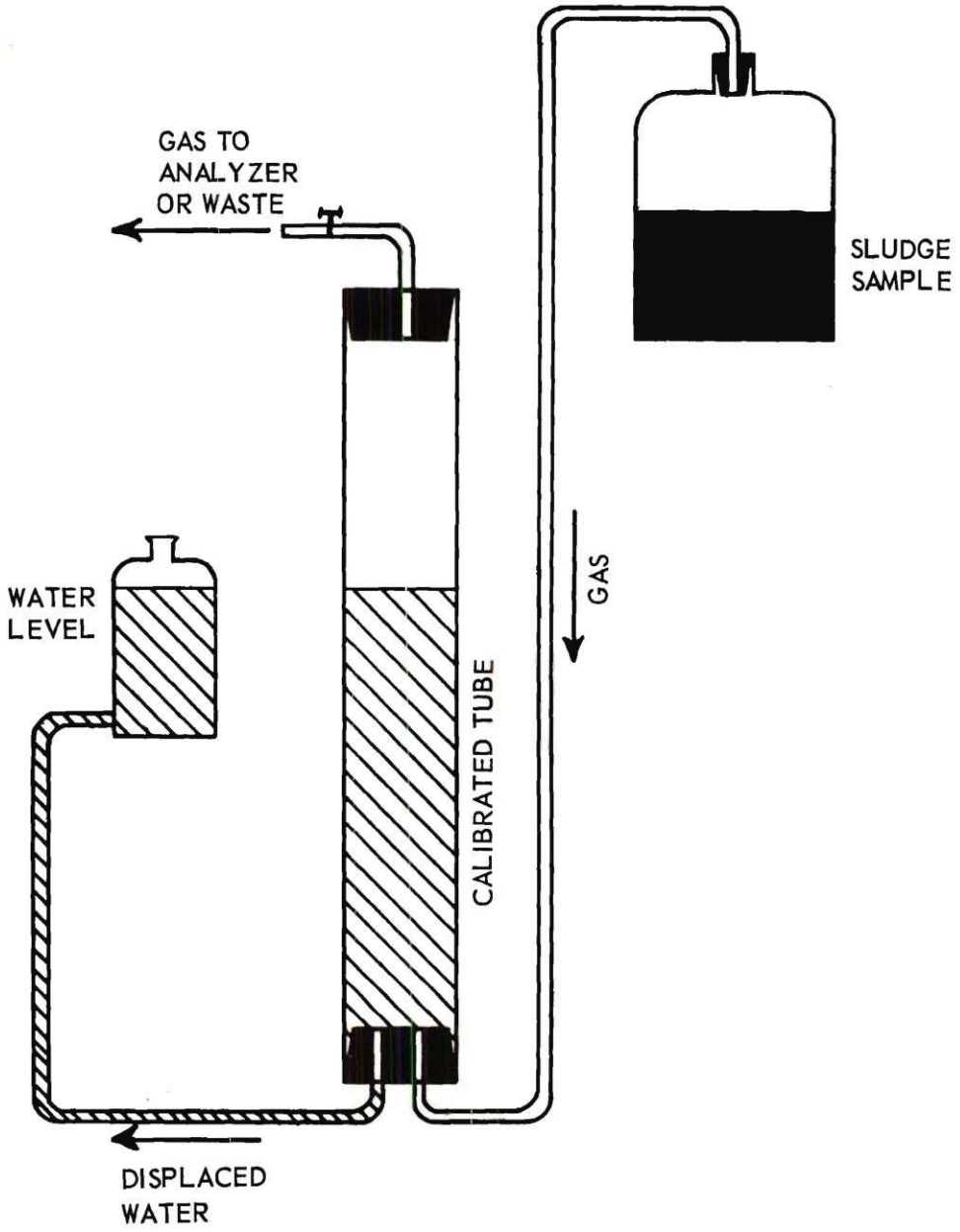

Figure 1. Diagram of Apparatus

The method employed in this study took advantage of the phenomenon of surface tension by the use of a small opening (about three-sixteenths inch) at the bottom of the calibrated tube. (The figure shows the apparatus assembled for taking a reading; the displaced water of a 24-hour period actually flowed to waste through a short tube.) The pressure due to the unremoved water and the introduced gas then equaled the pressure of the atmosphere. It follows that the volume of the gas in the tube depended upon the pressure of the atmosphere, according to the basic law of Boyle.

A bottle was connected to the bottom of the calibrated tube at the time of the reading by means of flexible tubing. The level of the water in the bottle could then be adjusted to match the level of the water in the calibrated tube, thus balancing the effect of the pressure due to the water column in the tube. The pressure of the gas in the tube would then be identical to the pressure of the atmosphere. The gas pressure in the tube included the pressure due to water vapor, the magnitude of which could be obtained from a handbook. A measurement of barometric pressure and of gas temperature permitted the employment of the laws of Boyle and of Charles and Gas-Lussac to calculate the gas volume under standard or comparable conditions.

The calibrated tube was refilled after each daily reading by releasing the clamp at the top and raising the connected bottle, thereby filling the calibrated tube with water and forcing out the gas. The tube was then sealed by the clamp and was thus made ready for the gas production of the next day.

CHAPTER III

PROCEDURE

The experimentation consisted of comparing the quantities of gas produced daily by fermenting aliquots of a mixture of raw and digested sludge, each containing varying amounts of potassium chromate. Variables of primary consideration, other than the concentration of chromate, were temperature and the time at which the chromate was added.

Except for runs in which temperature was a variable, the samples were maintained at room temperature with year-round thermostatic control for human comfort. The mean room temperature varied with the season: winter, about 22°C.; spring, about 23°C.; and summer, about 25°C. Temperature and gas readings were taken at approximately noon each day.

In order that the effect of temperature might be studied, incubators were employed. Three temperatures used were room temperature, 30°C. and 37°C. Sample bottles were placed in the incubators, and the gas was collected at room temperature.

Each sample was agitated once daily by hand shaking. This was necessary to prevent the partial separation of the solids from the liquid phase due to sedimentation or sludge rising.

All chemicals added were first dissolved in small amounts of water to minimize high concentrations in localized portions of the samples. Also, the samples were agitated immediately after such additions.

Anaerobiosis was maintained by sealing the stoppers in the sample

bottles. The only addition of atmospheric oxygen may have occurred during the addition of chemicals after the run had started. Positive gas pressure in the sample bottles prevented oxygen from entering even if leaks were present.

Several preliminary experiments were performed to determine the sample, or aliquot, size and the ratio of digested sludge to raw sludge, such that the digestion would be completed in a reasonable length of time and with the production of a suitable daily quantity of gas. The sample size which permitted the production of the desired one liter of gas per day was found to be that amount of sample which contained 12 grams of organic matter in the raw sludge. The ratio of digested sludge to raw sludge which induced the digestion to be completed in two to three weeks was ascertained to be two parts of organic matter from the digested sludge to one part of organic matter from the raw sludge. An individual sample then contained 12 grams of organic matter from the raw sludge and 24 grams of organic matter from the digested sludge. The total weight of the sample was usually about 1,000 grams, depending to a great extent upon the quantity of water present. A sample calculation of the determination of the sample size is presented in Appendix I-A.

The content of solid matter in the sludge was found by placing a known quantity of sludge in an oven at 105°C. for 24 hours, cooling the dried sludge in a desiccator, and weighing it. The dried sludge was then put in a muffle furnace and ashed at a temperature at which the ashing dish glowed red. The organic content was taken as the difference in weight between the dry sludge and the ash.

Eight runs were set up in which potassium chromate was added in

varying amounts to several samples with other factors varied in a run. However, only seven runs were carried out satisfactorily; one run gave no reportable results, because the organisms of the digested sludge seed were deprived of food for too great a length of time before the run was set up. The effect of temperature was studied in two of the runs. The effect of varying the time at which the chromate was added was also studied in two runs. The effect of chromic ions was compared to the effect of chromate ions in two runs as a supplementary study. The concentrations of chromium were based on the ratio of chromium to the total sludge in a digestion bottle and reported as parts per million (p.p.m.). In the final run a study was made of the effect of varying the concentration of organic matter in the sample and also of varying the ratio of digested sludge to raw sludge. In all runs except the first run, duplicates were made of a few samples in order to permit the determination of the pH without disturbing the collection of gas. A tabulation of the quantities of materials contained in each sample, along with the conditions compared, are presented for each run in Appendix II.

CHAPTER IV

DISCUSSION OF RESULTS

The results of Run I indicated that 1,000 p.p.m. chromium[1] only temporarily retarded gas production as compared to the control while concentrations below 1,000 p.p.m. did not noticeably affect the digestion. As indicated in Figure 2, the rate of gas production of the aliquot containing 1,000 p.p.m. chromium surpassed that of the control during the final days of the run. (The curves presented in each figure were estimated by sight. The points are not shown on the figures but may be obtained by referring to the data of Appendix III.) Because of an error in calculations, the chromate was added in two doses about 16 hours apart. The yellow color of chromate disappeared within 24 hours after the second addition of chromate in all of the samples of this run.

In contrast to these results, 100 p.p.m. chromium retarded the gas production considerably in Run II, while 1,000 p.p.m. chromium allowed no gas production during 31 days of observation. The results from two samples containing chromium concentrations which allowed appreciable gas production are shown in Figure 3. The aliquot containing 400 p.p.m. chromium produced very little gas until the yellow color of chromate had disappeared. The disappearance of the yellow color of chromate followed by the recovery of the digestion, either partial or complete, was typical of samples, which were considerably, but not

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[1]Unless otherwise indicated, the chromium was added in each run as potassium chromate.

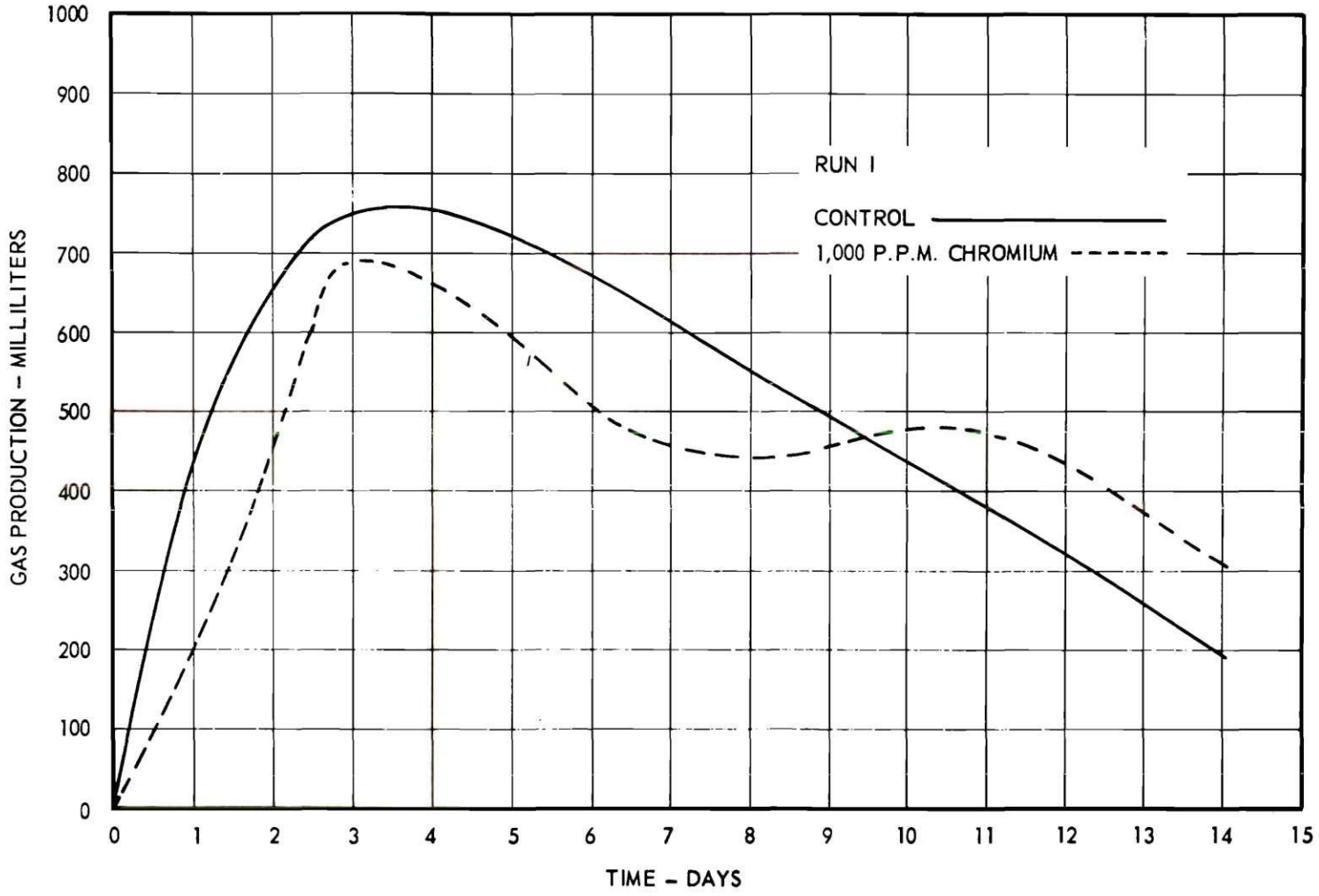

Figure 2. Effect of 1,000 p.p.m. Chromium on Gas Production in Run I

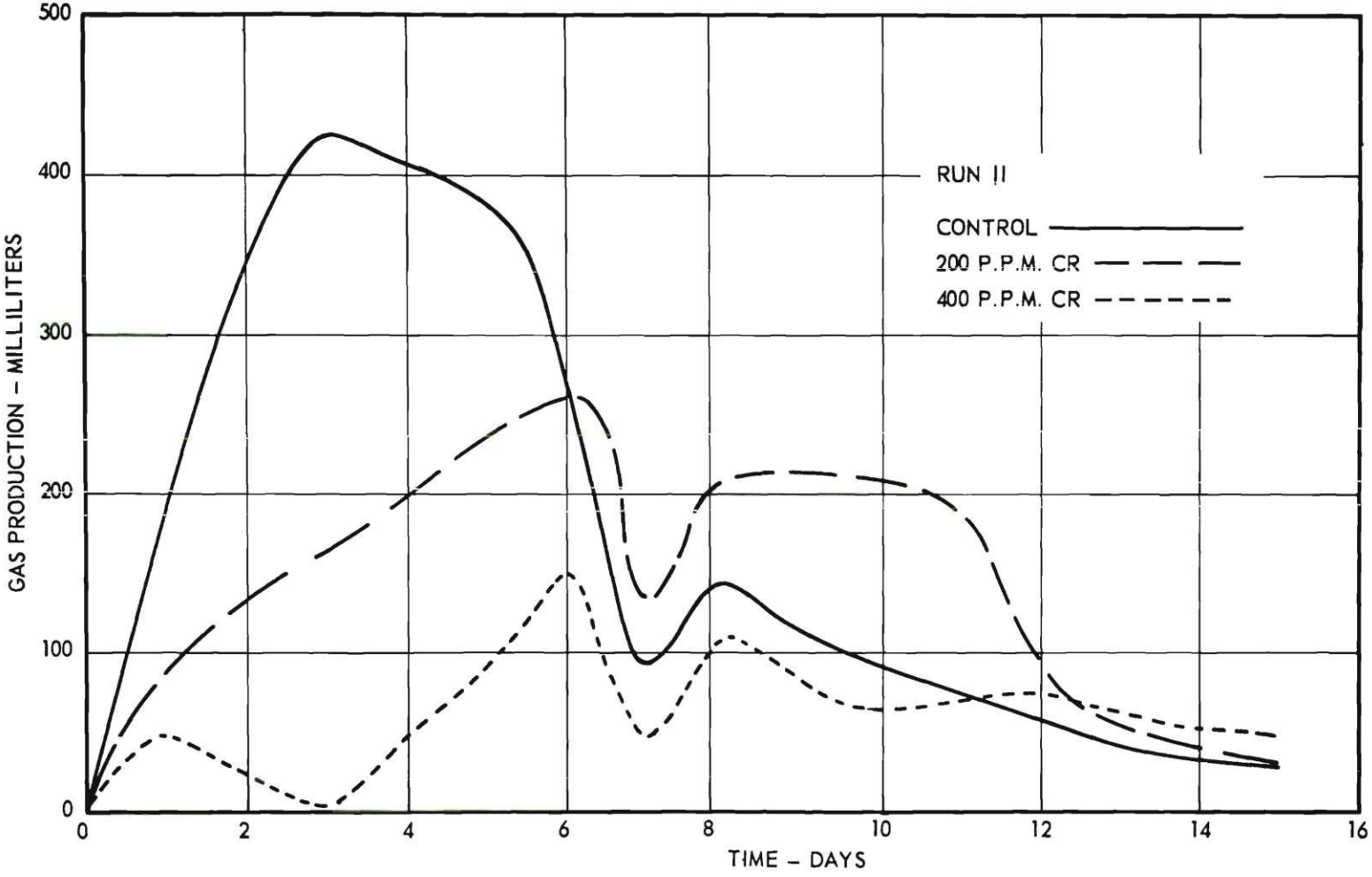

Figure 3. Effect of 200 p.p.m. and 400 p.p.m. Chromium on Gas Production in Run II

completely inactivated, throughout all of the runs. It was also typical of such samples that their rate of gas production surpassed that of the control during the last days that each run was observed. This may be verified by referring to the date of Appendix III which is a tabulation of the daily gas production of all of the aliquots of each run. It may also be observed from a study of these data that in each run the chromium caused a supressed maximum in the rate of gas production. This was accompanied by a prolonged period of gas production at a lower level than the control up to the point at which the rate of the control dropped off rapidly.

The effects of temperature and the time of chromate addition were studied in Run III. Figure 4 shows the daily rate of gas production of three samples containing 400 p.p.m. chromium, each of which was held at a different temperature during digestion: 22°, 30° and 37°C. It may be observed that the peak gas production was obtained sooner at the higher temperatures and that the total gas production was greater during the three-week period with each increase in temperature.

Figure 5 presents the results of adding 400 p.p.m. chromium to several different samples on different days during the digestion. It may be seen that the addition of the chromate during the period of highest activity resulted in better digestion than when added initially or after the peak of gas production. Each sample, following the addition of chromate, showed a marked depression in rate of gas production immediately and had a peak of activity after a period of recovery. (Values above 1,000 milliliters of gas per day were estimated from similar samples which did not exceed the maximum reading of the equipment.)

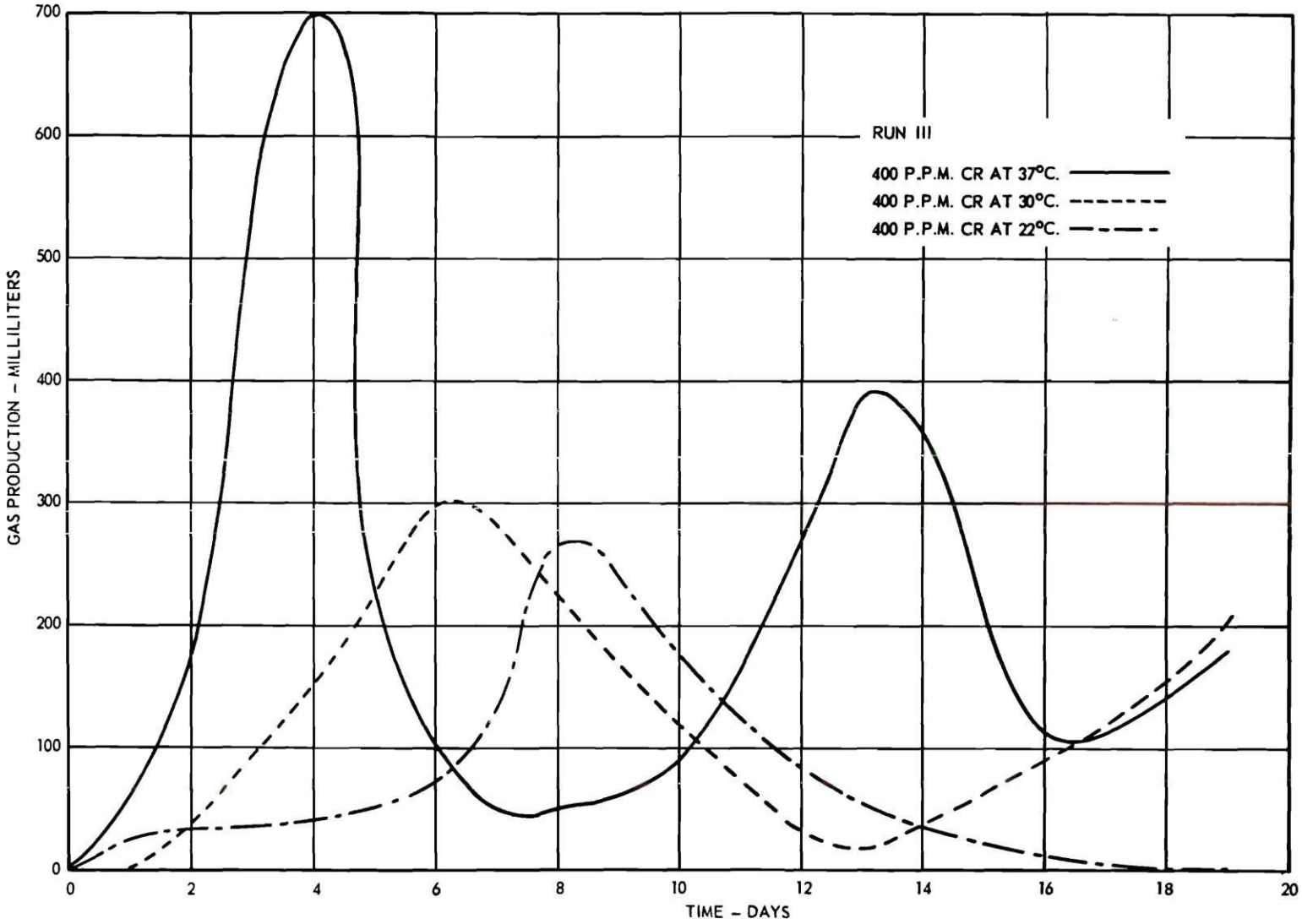

Figure 4. Comparison of Gas Productions by Samples Containing 400 p.p.m. Chromium at Three Different Temperatures in Run III

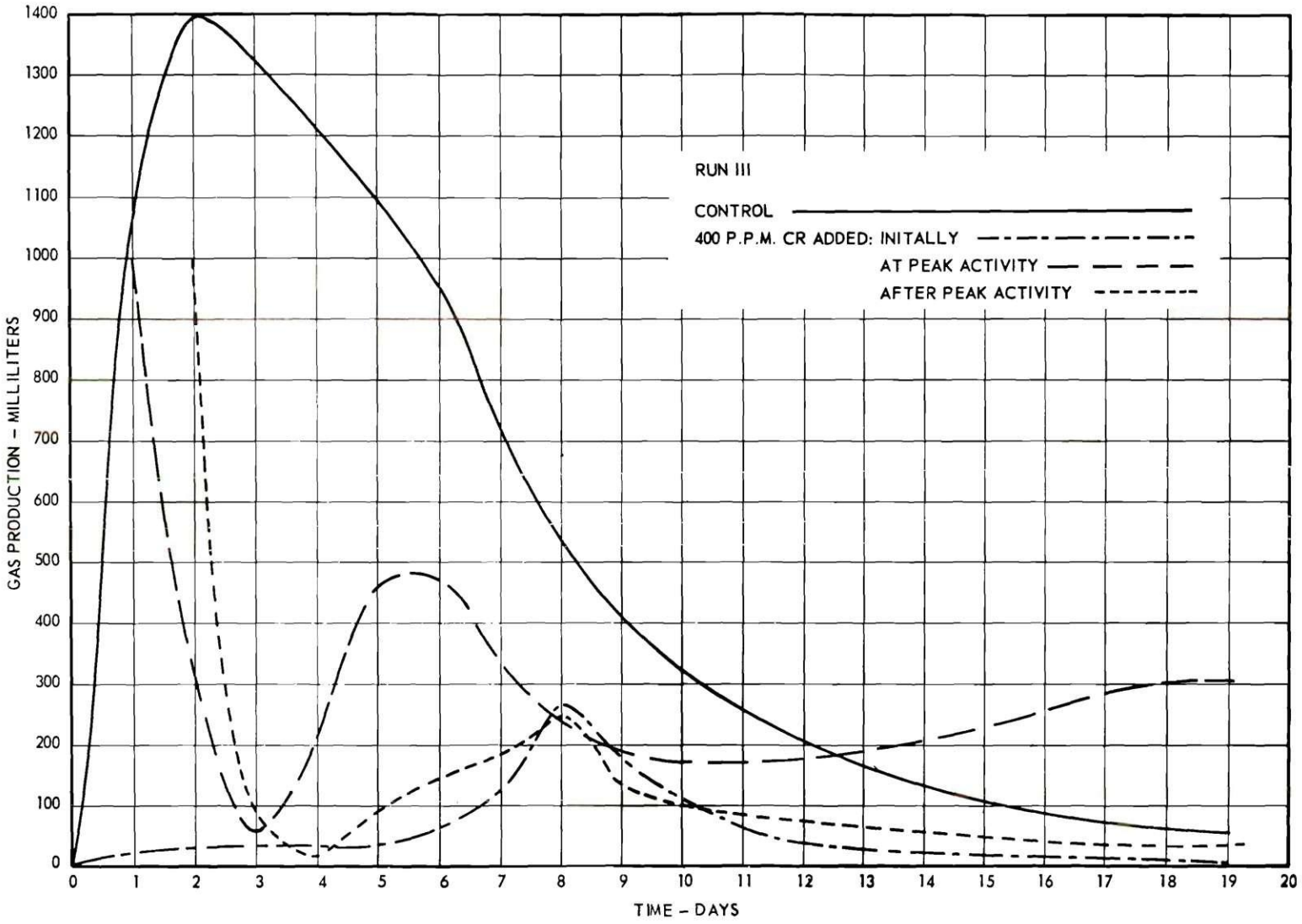

Figure 5. Comparison of Effects on Gas Production of Adding 400 p.p.m. Chromium at Different Times During Digestion in Run III

Run IV consisted of a further study of the effect of temperature. The concentration of solid matter in the sludge was very low, and the toxicity of the chromate was very pronounced. 200 p.p.m. chromium retarded the gas production seriously at each of the temperatures employed, with the result that no reportable comparison of toxicities at various temperatures was obtained.

The investigation in Run V was concentrated on determining the effect of adding chromate over a period of several days rather than in one dose. The curves of Figure 6 present a comparison of the results of adding 400 p.p.m. chromium initially, adding 50 p.p.m. chromium daily for seven days, and adding 100 p.p.m. chromium daily for seven days. It may be observed that the addition of the chromate before the digestion had begun resulted in a digestion which only slightly recovered, whereas the addition of chromate over the period of peak activity allowed the gas production to proceed at a higher rate, even after the addition of 700 p.p.m. chromium in one sample. Reference to the data of Appendix III will show that the addition of chromate over a period of several days beginning with the second day (Samples 10 and 11) resulted in a more active digestion than when the daily additions were begun before the digestion had begun (Samples 8 and 9).

Run IV was designed to compare the effects of chromic ions and chromate ions. Although it appeared that the chromic ions were less toxic than the chromate ions, the effect of the nitrate ions, from the chromic nitrate used, interfered with the digestion in such a way that a definite comparison could not be made.

In order to study the relative toxicities of chromic ions and

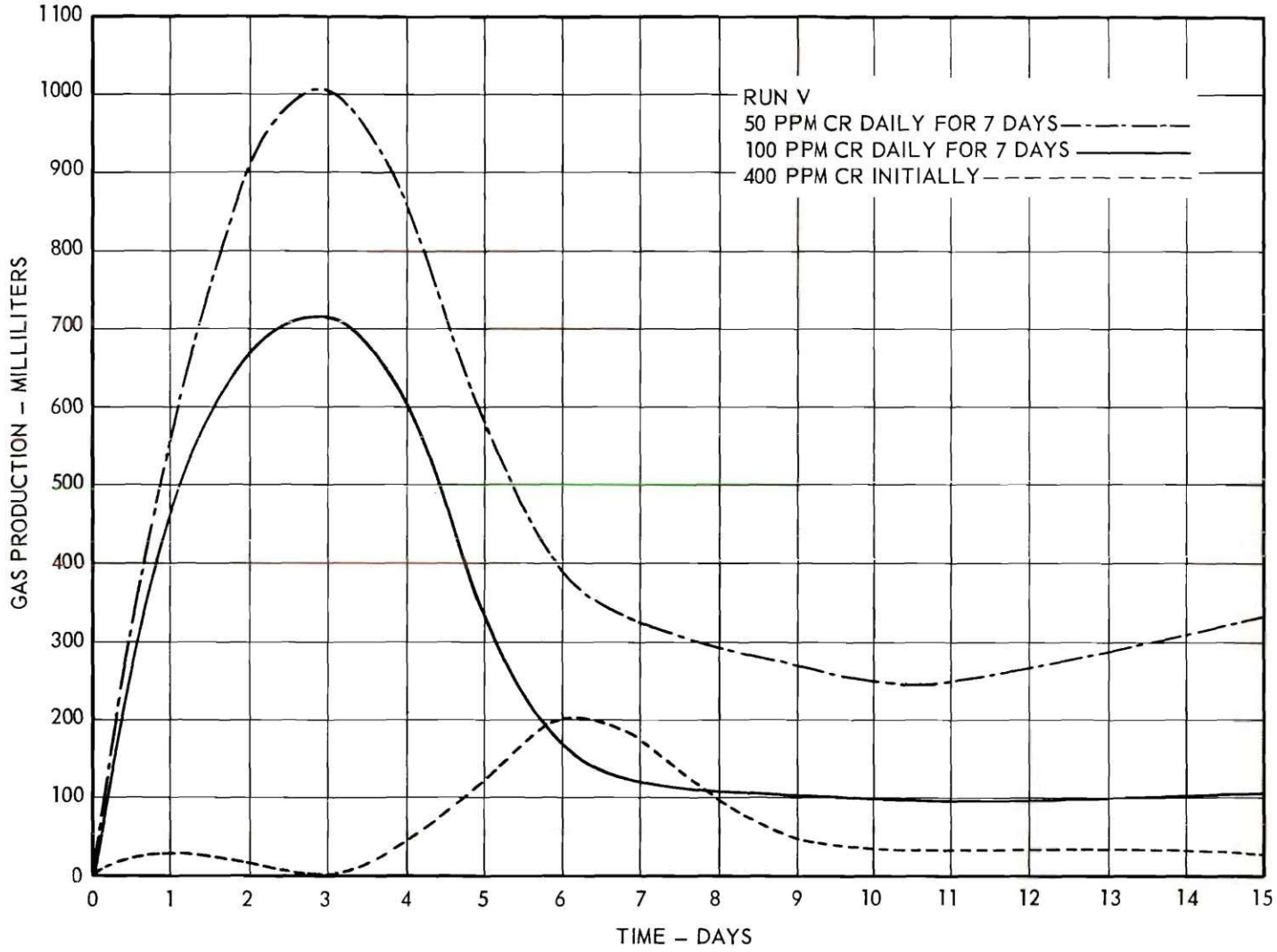

Figure 6. Comparison of Gas Productions of Samples with 400 p.p.m. Chromium Added Initially, 50 p.p.m. Chromium Added Daily and 100 p.p.m. Chromium Added Daily in Run V

chromate ions further, Run VII was set up using chromic chloride as the source of chromic ions. Figure 7 shows that the effect of the chromic ions was much less than that of the chromate ions, even though the chromic ions themselves allowed less than half the gas production of the control in this case. Even though 400 p.p.m. trivalent chromium allowed only about 10 per cent of the gas production of the control, 400 p.p.m. hexavalent chromium allowed no appreciable gas production during two weeks of observation.

One sample of Run VII was diluted with about 40 per cent additional water before adding the chromate in order to study the relation of the concentration of organic matter in the sludge to the toxicity of the chromate. The diluted sample, containing 300 p.p.m. chromium based on the total volume of sludge, produced about three-fourths of the amount of gas produced by an undilited sample containing 300 p.p.m. chromium. A comparison of several runs is made in Table 1 with respect to organic content and toxicity. Although a different sludge was used in each run, there is an apparent trend toward increasing toxicity with increasing dilution. There is a probability that the ratio of chromium to organic content is an important factor in this relation.

The variation in toxicity exhibited by corresponding concentrations of chromate between different runs in this investigation was in accordance with the wide divergence of toxicities reported in the literature. An analysis of the results of this study indicates that the biological condition of the sludge is of primary importance in determining the toxicity of chromate on the digestion process. An increase in biological activity, then, brought about a decrease in the effect of the chromate.

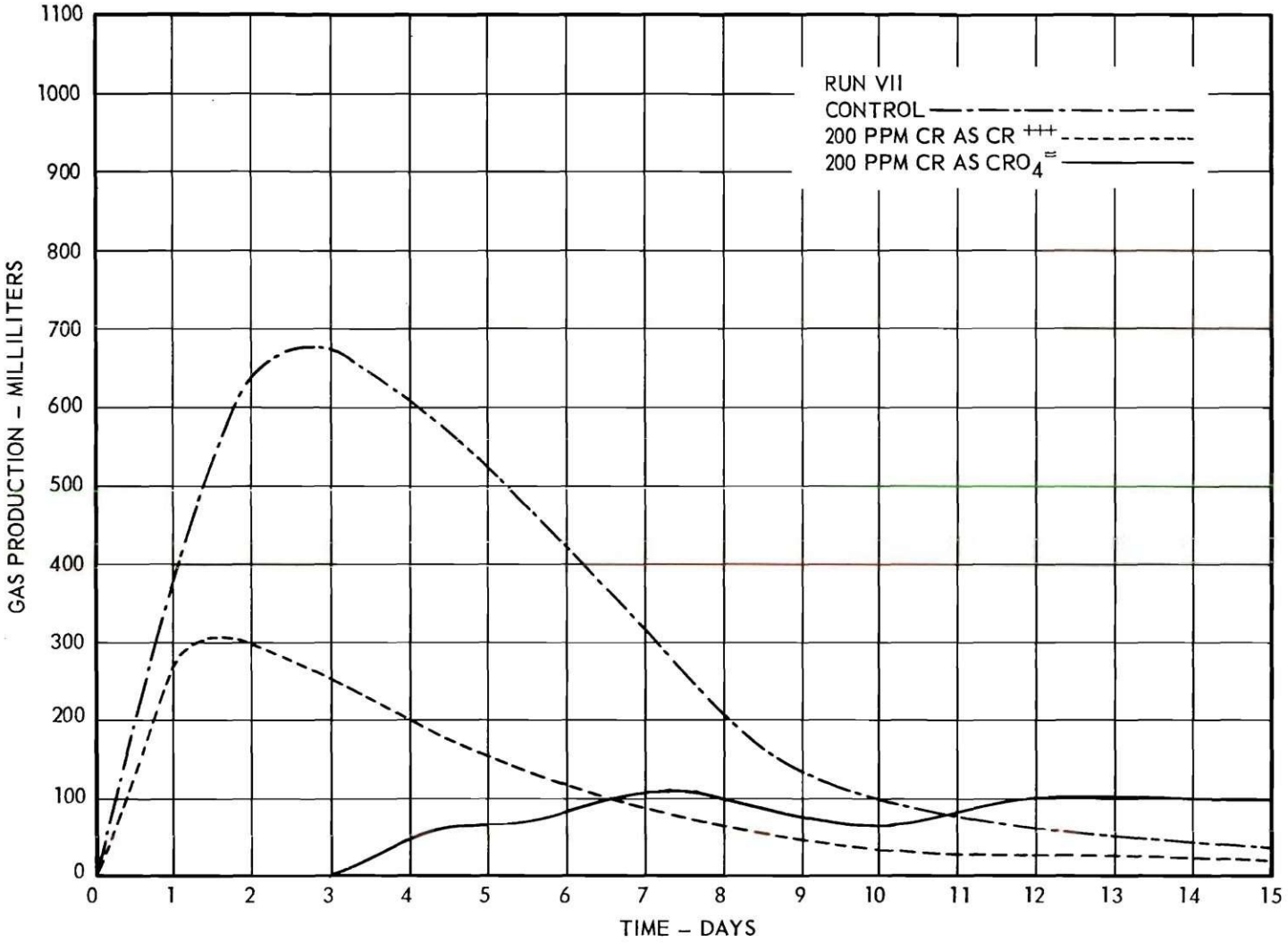

Figure 7. Comparison of Effects on Gas Production of 200 p.p.m. Hexavalent Chromium and 200 p.p.m. Trivalent Chromium in Run VII

Table 1. Relation of Organic Content of Sludge to Effect of Chromate on Sludge Digestion

Run	Sample Size (Grams)	Organic Content (Per Cent)	Chromium Concentration (p.p.m.) based on Total Sludge	Chromium Concentration (p.p.m.) based on Organic Content	Gas Production (Per Cent of Control, 14 days)
I	750	4.8	200	4,200	91
II	750	4.8	200	4,200	87
V	1,010	3.6	200	5,600	57
VI	1,060	3.4	200	5,900	68
VII	1,510	2.4	200	8,400	21
IV	2,050	1.8	200	11,400	5

Increases in digestion activity were effected in this study by increasing the temperature and by allowing the organisms of digestion to flourish before adding the chromate. The disagreement in toxicity reported in the literature may be explained by the fact that the biological condition of the sludge was different in each study.

The ability of the organisms to reduce the chromate was essential to the recovery of the digestion process. The recovery of the digestion only after the disappearance of the yellow color of chromate supports the statement by Hoover (4) that the persistent yellow color of chromate is objectionable to sludge digestion. The incomplete recovery of most samples is believed to be attributed to the high concentration of chromic ions remaining when the chromate was reduced. The fact that the samples which produced no significant gas also reduced the chromate may be ascribed to the fact that the methane organisms were compeletly inactivated by the initial high concentration of chromate ions or, again, to the fact that the resulting high concentration of chromic ions was very toxic.

A review of the relative toxicities disclosed in this investigation suggests that 200 p.p.m. chromium should be the limiting concentration allowed in an anaerobic digestion tank in one infrequent dose. As pointed out, the biological condition of the sludge is the final factor defining any variation about the above value.

CHAPTER V

CONCLUSIONS

An increase in the activity of an anaerobic digestion of sewage sludge will result in a decrease in the toxicity of chromate to that digesting mixture.

The recovery, either partial or complete, of a methane fermentation of sewage sludge, to which chromate has been added, is only effected after the chromate has been reduced.

A decrease in the concentration of organic matter in a sewage sludge will result in an increase in the deleterious effect of a given concentration of chromate in an anaerobic digestion of that sludge.

Chromate ions are more toxic to the anaerobic digestion of sewage sludge than are chromic ions, based on the same amount of chromium.

200 p.p.m. hexavalent chromium should be the maximum concentration allowed in an anaerobic digestion tank in one infrequent dose in a sewage treatment plant.

CHAPTER VI

RECOMMENDATIONS

The present investigation has uncovered several factors which, it is recommended, should be studied further for a deeper comprehension of the effect of chromate on the anaerobic digestion of sewage sludge. Further study of the relation of the concentration of organic matter in a sewage sludge to the effect of chromate on the digestion of that sludge might disclose a more well-defined relation between the two factors.

Numerous comparisons of analyses of gas produced by samples contaninint various concentrations of chromate, as previously made by Barnes and Braidech (6), may prove valuable.

Varying the ratio of digested sludge to raw sludge may be a method of changing the activity of the digestion in such a way that the effect of chromate on the digestion would be changed.

Studies of the effect of chromate on digestions in the thermophillic range of temperature might prove informative, since thermophillic digestion may be employed to a larger extent as experience in that field is acquired.

Since the anaerobic digestion of sewage sludge is sensitive to changes in pH, an intensive study of the changes in pH over periods of digestion in the presence of chromate would probably reveal further insight into understanding the effect of chromate on the digestion process.

A P P E N D I C E S

Appendix I. Sample Calculations

A. Determination of Sample Size (Data from Run V)

Weight of	Raw Sludge (Grams)	Digested Sludge (Grams)
dish and sludge	108.5	106.6
dish	52.01	46.61
sludge	56.5	60.0
dish and dry sludge	55.28	50.67
dish	52.01	46.61
dry sludge	3.27	4.06
dish and ash	53.17	48.58
dish	52.01	46.61
ash	1.16	1.97
dry sludge	3.27	4.06
ash	1.16	1.97
organic matter	2.11	2.09
percentage of organic matter in the sludge	$\frac{2.11}{56.5} = 3.74\%$	$\frac{2.09}{60.0} = 3.48\%$

Raw sludge

$$\frac{3.74 \text{ g organic matter}}{100 \text{ g raw sludge}} = \frac{12 \text{ g organic matter}}{x}$$

$$x = 320 \text{ g raw sludge}$$

Digested sludge

$$\frac{3.48 \text{ g organic matter}}{100 \text{ g digested sludge}} = \frac{24 \text{ g organic matter}}{y}$$

$$y = 690 \text{ g digested sludge}$$

Sample size = 320 g plus 690 g = 1,010 g

B. Determination of Quantity of Potassium Chromate Added to Sample
(Data from Run V)

Molecular weight of $K_2 CrO_4$	194.2
Atomic weight of Cr	52.0

$$\frac{194.2}{52.0} = 3.74 \frac{g\ K_2 CrO_4}{g\ Cr}$$

Basis: 1,000 p.p.m. Cr in Sample

$$1{,}000 \text{ p.p.m. Cr} = \frac{1.01 \text{ g Cr}}{1{,}010 \text{ g Sample}}$$

Weight of $K_2 CrO_4$ = (1.01 g Cr) (3.74 g $K_2 CrO_4$/g Cr)

= 3.78 g $K_2 CrO_4$

C. Determination of Decrease in Organic Content of a Sample During Digestion (Data from Run V)

Weight of	Control (Grams)
dish and sludge	120.5
dish	52.01
	68.5
dish and dry sludge	55.55
dish	52.01
dry sludge	3.54
dish and ash	53.96
dish	52.01
ash	1.95
dry sludge	3.54
ash	1.95
organic matter	1.59
percentage of organic matter in the sludge	1.59 / 68.5 = 2.32%
Sample size	1,010 g
Water added	25
Total weight	1,035 g

(1,035 g) (0.0232) = 24.0 g organic matter

The sample contained 12 grams of raw organic matter and 24 grams of digested organic matter before digestion. Assuming the digested organic matter to have been biologically stable, the 12 grams of raw organic matter had been completely converted to gas.

Appendix II. Contents of Samples as Set Up in Each Run

Run I. December 2, 1955

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium (p.p.m.)	Potassium Chromate (Grams)
1	265	485	12	24	0	0
2	265	485	12	24	1	0.0028
3	265	485	12	24	5	0.0140
4	265	485	12	24	10	0.0380
5	265	485	12	24	20	0.0560
6	265	485	12	24	50	0.140
7	265	485	12	24	100	0.280
8	265	485	12	24	200	0.560
9	265	485	12	24	500	1.40
10	265	485	12	24	1,000	2.80

Run II. February 3, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium (p.p.m.)	Potassium Chromate (Grams)
1	270	480	12	24	0	0
2	270	480	12	24	100	0.278
3	270	480	12	24	200	0.556
4	270	480	12	24	400	1.11
5	270	480	12	24	700	1.95
6	270	480	12	24	1,000	2.78
7	270	480	12	24	2,000	5.56
8	270	480	12	24	4,000	11.1
9	270	480	12	24	7,000	19.5
10	270	480	12	24	10,000	27.8

Run III. March 8, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium (p.p.m)	Potassium Chromate (Grams)	Date Chromate Added	Temperature of Digestion (°C.)
1	290	750	12	24	0	0	3-8	22
2	290	750	12	24	400	1.55	3-8	22
3	290	750	12	24	1,000	3.88	3-8	22
4	290	750	12	24	400	1.55	3-9	22
5	290	750	12	24	400	1.55	3-10	22
6	290	750	12	24	400	1.55	3-11	22
7	290	750	12	24	400	1.55	3-12	22
8	290	750	12	24	400	1.55	3-13	22
9	145	375	6	12	0	0	3-8	37
10	145	375	6	12	400	0.776	3-8	37
11	145	375	6	12	1,000	1.94	3-8	37
12	145	375	6	12	0	0	3-8	30
13	145	375	6	12	400	0.776	3-8	30
14	145	375	6	12	1,000	1.94	3-8	30

Run IV. April 3, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium (p.p.m.)	Potassium Chromate (Grams)	Temperature of Digestion (°C.)
1	1,360	690	12	24	0	0	23
2	1,360	690	12	24	100	0.765	23
3	1,360	690	12	24	200	1.53	23
4	1,360	690	12	24	400	3.06	23
5	680	345	6	12	0	0	30
6	680	345	6	12	200	0.765	30
7	680	345	6	12	400	1.53	30
8	680	345	6	12	0	0	32
9	680	345	6	12	200	0.765	32
10	680	345	6	12	400	1.53	32

Run V. April 26, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium at Start (p.p.m.)	Potassium Chromate at Start (Grams)	Additional Potassium Chromate Added on Date Indicated (p.p.m. Cr) April 27	April 28	April 29	April 30	May 1	May 2
1	320	690	12	24	0	0	0	0	0	0	0	0
2	320	690	12	24	50	0.189	0	0	0	0	0	0
3	320	690	12	24	100	0.378	0	0	0	0	0	0
4	320	690	12	24	200	0.756	0	0	0	0	0	0
5	320	690	12	24	400	1.51	0	0	0	0	0	0
6	320	690	12	24	800	3.02	0	0	0	0	0	0
7	320	690	12	24	1,000	3.78	0	0	0	0	0	0
8	320	690	12	24	50	0.189	50	50	50	50	50	50
9	320	690	12	24	100	0.378	100	100	100	100	100	100
10	320	690	12	24	0	0	50	50	50	50	50	50
11	320	690	12	24	0	0	100	100	100	100	100	100
12	320	690	12	24	0	0	50	0	50	0	50	0
13	320	690	12	24	0	0	100	0	100	0	100	0
14	320	690	12	24	0	0	0	100	0	0	100	0

Run VI. May 24, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium as Ions Indicated (p.p.m.) Chromate	Chromic	Potassium Chromate (Grams)	Chromic Nitrate Nonahydrate (Grams)
1	310	750	12	24	0	0	0	0
2	310	750	12	24	100	0	0.392	0
3	310	750	12	24	200	0	0.785	0
4	310	750	12	24	300	0	1.18	0
5	310	750	12	24	400	0	1.57	0
6	310	750	12	24	800	0	3.14	0
7	310	750	12	24	75	225	0.294	1.85
8	310	750	12	24	150	150	0.588	1.23
9	310	750	12	24	225	75	0.882	0.615
10	230	560	8	18	0	0	0	0
11	310	950[a]	12	22	0	0	0	0
12	310	950[b]	12	23	200	0	0.942	0
13	310	750	12	24	0	300	0	2.46
14	310	750	12	24	0	0	0	0[c]

[a]Digested sludge from Sample 1 of Run V.
[b]Digested sludge from Sample 3 of Run V.
[c]Sample 14 contained 1.86 grams of Potassium Nitrate, an amount of nitrate equivalent to that in Sample 13.

Run VII. June 22, 1956

Sample	Raw Sludge (Grams)	Digested Sludge (Grams)	Raw Organic Matter (Grams)	Digested Organic Matter (Grams)	Chromium as Ions Indicated (p.p.m.) Chromate	Chromic	Potassium Chromate (Grams)	Chromic Chloride Hexahydrate (Grams)
1	650	860	12[b]	24	0	0	0	0
2	650	860	12	24	100	0	0.565	0
3	650	860	12	24	200	0	1.13	0
4	650	860	12	24	300	0	1.70	0
5	650	860	12	24	400	0	2.26	0
6	650	860	12	24	800	0	4.52	0
7	650	860	12	24	1,000	0	5.65	0
8	650	860	12	24	0	200	0	1.55
9	650	860	12	24	0	400	0	3.10
10	650	860	12	24	0	800	0	6.20
11[a]	650	860	12	24	0	0	0	0
12[a]	650	860	12	24	300	0	2.13[c]	0
13	430	860	8	24	0	0	0	0
14	430	860	8	24	300	0	1.45	0

[a]Samples 11 and 12 contained 390 milliliters more water than did the other samples. This water was added to study the effect of varying the concentration of organic matter.

[b]One gram of filter paper was added to samples 1 through 9 on July 2 in order to determine whether the samples had recovered and needed only additional organic matter to produce gas.

[c]Although sample 12 contained the same concentration of chromium (300 p.p.m.) as did sample 4, sample 12 contained a larger quantity of potassium chromate since it contained more water. (See note [a].)

Appendix III. Daily Gas Production

Run I. December 2, 1955, 4:00 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 22° C. and 74 cm. Mercury, including water vapor)				
		1	2	3	4	5
12-3	10:00 A.M.	435	485	500	475	515
12-4	12:30 P.M.	640	750	730	655	790
12-5	1:00 P.M.	780	875	865	825	850
12-6	10:00 A.M.	605	690	690	625	700
12-7	12:00 Noon	810	825	885	840	895
12-8	10:00 A.M.	665	755	675	675	780
12-9	1:00 P.M.	700	690	685	665	740
12-10	10:00 A.M.	420	395	400	400	425
12-11	12:30 P.M.	625	560	560	550	555
12-12	1:00 P.M.	500	400	415	400	425
12-13	10:30 A.M.	360	280	290	300	285
12-14	12:30 P.M.	380	275	285	285	285
12-15	10:00 A.M.	275	190	185	185	210
12-16	12:30 P.M.	185	140	145	135	135
14-day Total		7,380	7,310	7,310	7,015	7,590

Run I - continued

6	7	8	9	10
440	-	425	335	200
720	745	540	540	450
760	870	650	800	725
605	690	545	600	555
825	870	600	745	625
720	680	500	630	535
675	700	525	660	555
375	400	340	410	395
525	585	520	650	630
350	425	465	500	525
270	300	420	405	460
275	290	470	445	500
250	185	300	300	365
150	150	420	250	300
6,940	-	6,720	7,270	6,820

Run II. February 3, 1956, 5:30 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 22° C. and 74 cm. Mercury, including water vapor)				
		1	2	3	4	5
2-4	12:30 P.M.	175	125	100	50	25
2-5	2:30 P.M.	350	225	130	25	35
2-6	1:00 P.M.	425	265	170	0	0
2-7	10:00 A.M.	370	260	175	50	30
2-8	12:00 Noon	380	300	245	80	0
2-9	1:30 P.M.	280	315	260	150	0
2-10	10:00 P.M.	90	175	135	45	0
2-11	12:00 Noon	145	260	235	110	0
2-12	2:00 P.M.	95	190	210	70	0
2-13	1.00 P.M.	65	120	205	65	0
2-14	12:30 P.M.	75	115	195	75	0
2-15	3:00 P.M.	60	100	75	80	0
2-16	10:30 A.M.	35	40	60	40	0
2-17	10:00 A.M.	35	45	50	50	0
2-18	11:00 A.M.	55	55	55	60	0
2-19	4:00 P.M.	55	65	40	65	0
2-20	1:00 P.M.	25	30	10	25	0
2-21	12:00 Noon	15	15	10	15	0
2-22	12:00 Noon	35	50	20	50	0
2-23	10:30 A.M.	60	80	50	60	0
2-24	10:30 A.M.	65	90	35	50	0
2-25	11:00 A.M.	115	125	75	75	30
2-26	2:30 P.M.	55	55	60	65	5
2-27	1:00 P.M.	50	75	95	80	30
2-28	10:30 A.M.	35	65	85	75	30
2-29	12:00 Noon	25	40	35	100	0
3-1	11:00 A.M.	55	55	60	150	35
3-2	10:30 A.M.	55	55	75	200	50
3-3	11:30 A.M.	55	50	85	210	45
3-4	12:30 P.M.	30	25	40	130	10
3-5	1:00 P.M.	75	60	70	100	55
14-day Total		2,580	2,535	2,245	890	95

Note: Samples 6 through 10 gave no significant gas production.

Run III. March 8, 1956, 3:00 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 22° C. and 74 cm. Mercury, including water vapor)					
		1	2	3	4	5	6
3-9	11:00 A.M.	1,000	35	45	1,000	-	1,000
3-10	12:30 P.M.	1,000	35	0	290	1,000	1,000
3-11	2:30 P.M.	1,000	15	0	65	80	1,000
3-12	1:00 P.M.	1,000	45	0	230	20	60
3-13	9:30 A.M.	1,000	15	0	460	95	0
3-14	11:30 A.M.	1,000	70	0	475	150	195
3-15	11:00 A.M.	700	110	15	280	175	155
3-16	11:00 A.M.	550	270	5	245	255	130
3-17	11:30 A.M.	390	170	25	185	130	105
3-18	12:30 P.M.	380	175	20	225	130	145
3-19	11:00 A.M.	225	35	20	170	70	120
3-20	1:00 P.M.	225	50	0	215	10	120
3-21	2:30 P.M.	190	35	0	245	0	135
3-22	11:00 A.M	125	20	0	195	0	100
3-23	11:00 A.M.	115	20	0	240	50	125
3-24	11:30 A.M.	95	20	0	250	50	120
3-25	-	-	-	-	-	-	-
3-26	12:30 P.M.	170	0	125	610	130	275
3-27	10:00 A.M.	55	15	60	295	50	125
14-day Total		8,785	1,080	130	4,280	-	4,265

Run III - continued

7	8	9	10	11	12	13	14
700	1,000	775	190	25	380	0	0
1,000	1,000	700	0	0	770	95	85
1,000	1,000	395	55	0	1,000	0	0
650	1,000	470	360	0	950	20	0
70	1,000	540	105	0	515	75	0
0	35	285	25	0	480	175	0
0	50	170	25	0	275	100	0
0	100	185	25	0	225	100	0
30	60	125	0	0	150	25	0
100	60	140	60	0	150	65	0
95	35	30	40	0	85	20	0
120	45	75	-	0	75	20	35
130	85	45	205	0	70	0	70
95	45	0	95	0	45	25	25
115	50	30	110	0	75	35	20
120	70	10	25	0	70	50	20
-	-	-	-	-	-	-	-
285	150	0	80	0	75	125	25
125	65	0	55	0	50	110	20
14-day Total							
3,990	5,515	3,935	1,555	25	5,170	720	215

Run IV. April 3, 1956, 5:00 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 22° C. and 74 cm. Mercury, including water vapor)				
		1	2	3	4	5
4-4	10:00 A.M.	-	80	0	0	325
4-5	11:00 A.M.	-	220	25	30	635
4-6	11:30 A.M.	-	275	0	0	590
4-7	10:30 A.M.	490	375	0	0	495
4-8	2:00 P.M.	1,000	385	0	-	740
4-9	10:30 A.M.	585	200	0	0	600
4-10	11:30 A.M.	750	220	40	5	775
4-11	10:00 A.M.	695	170	110	0	645
4-12	11:00 A.M.	770	155	80	0	525
4-13	8:00 A.M.	685	120	75	0	325
4-14	12:00 Noon	1,000	175	75	135	135
4-15	5:30 P.M.	1,000	185	60	180	200
4-16	11:30 A.M.	610	110	25	50	125
4-17	10:30 A.M.	910	120	5	45	85
4-18	11:00 A.M.	925	160	25	55	55
4-19	11:00 A.M.	765	155	30	40	30
4-20	8:00 A.M.	505	140	0	25	50
4-21	12:00 Noon	570	235	20	10	55
4-22	2:30 P.M.	350	260	20	30	40
4-23	11:00 A.M.	210	185	10	0	0
4-24	12:00 Noon	210	250	5	10	0
14-day Total		-	2,790	495	445	6,200

Run IV - continued

6	7	8	9	10
25	0	615	25	55
15	0	1,000	0	20
0	0	810	0	0
0	0	810	0	0
55	0	1,000	75	0
30	0	810	55	0
35	0	845	70	0
45	75	490	25	0
0	50	285	0	0
25	0	145	20	0
20	0	145	25	0
50	0	115	55	0
50	0	30	35	0
0	0	55	90	0
0	0	50	135	0
15	5	50	130	0
35	0	45	100	0
50	25	30	90	0
50	0	0	65	0
25	45	0	75	0
75	0	0	115	0
14-day Total				
350	125	7,155	475	75

Run V. April 26, 1956, 6:00 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measures at about 23° C. and 74 cm. Mercury, including water vapor)					
		1	2	3	4	5	6
4-27	11:00 A.M.	690	575	460	175	0	0
4-28	12:00 Noon	1,000	1,000	920	440	25	10
4-29	3:30 P.M.	1,000	1,000	1,000	675	20	10
4-30	10:30 A.M.	1,000	1,000	865	630	0	0
5-1	12:30 P.M.	1,000	1,000	1,000	750	65	0
5-2	10:30 A.M.	1,000	850	695	425	100	0
5-3	11:00 A.M.	1,000	950	735	435	200	0
5-4	11:00 A.M.	900	850	700	375	180	0
5-5	11:00 A.M.	860	860	725	370	85	0
5-6	1:00 P.M.	725	750	735	370	40	70
5-7	11:00 A.M.	530	580	600	325	40	100
5-8	11:00 A.M.	510	595	690	410	50	80
5-9	11:30 A.M.	450	530	705	460	30	50
5-10	11:30 A.M.	325	580	580	445	40	50
5-11	1:00 P.M.	310	340	525	560	35	90
5-12	1:00 P.M.	210	235	370	480	5	45
5-13	11:00 A.M.	150	160	230	400	20	40
5-14	2:30 P.M.	190	200	275	565	10	35
5-15	12:00 Noon	130	135	180	400	15	45
5-16	11:30 A.M.	90	85	130	370	25	50
5-17	11:30 A.M.	80	60	100	280	0	0
5-18	8:00 A.M.	70	55	90	225	0	0
5-19	1:30 P.M.	100	90	125	280	10	20
5-20	1:00 P.M.	60	60	75	150	15	0
5-21	11:00 A.M.	65	60	70	150	30	0
14-day Total		10,990	11,120	10,410	6,285	875	360

Run V. - continued

7	8	9	10	11	12	13	14
0	535	455	630	560	-	-	-
15	950	675	1,000	840	990	765	-
45	1,000	710	1,000	940	1,000	1,000	1,000
20	675	425	735	400	750	520	925
20	675	430	805	450	1,000	850	1,000
10	360	180	445	200	550	260	425
0	325	80	355	115	680	340	515
0	335	155	385	165	630	325	510
15	300	120	330	130	675	290	525
0	270	105	350	140	650	250	520
70	265	110	305	115	550	260	460
110	300	110	380	125	630	310	560
100	310	110	410	130	655	400	625
100	325	90	430	120	510	400	575
75	410	110	510	130	480	495	580
110	370	100	460	120	325	470	460
50	325	60	280	90	205	370	325
85	475	135	530	160	250	525	365
40	385	100	405	140	180	410	240
40	380	80	405	125	120	410	200
10	350	80	310	150	100	330	130
20	320	100	290	150	80	300	120
25	420	175	380	260	100	410	140
0	300	140	260	210	50	260	80
0	260	155	210	230	70	250	70
14-day Total							
505	6,625	3,755	7,560	4,430	-	-	-

Run VI. May 24, 1956, 7:30 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 24° C. and 74 cm. Mercury, including water vapor)					
		1	2	3	4	5	6
5-25	8:00 A.M.	335	-	0	0	0	0
5-26	12:00 Noon	850	-	275	0	0	0
5-27	1:00 P.M.	930	770	340	0	0	0
5-28	12:30 P.M.	860	700	420	0	0	0
5-29	11:30 A.M.	890	700	370	0	0	50
5-30	12:30 P.M.	875	730	350	10	10	0
5-31	11:30 A.M.	730	620	340	10	210	0
6-1	11:30 A.M.	590	680	420	10	140	0
6-2	12:00 Noon	290	450	360	0	40	0
6-3	12:30 P.M.	180	300	310	0	30	0
6-4	11:00 A.M.	160	250	310	50	35	0
6-5	12:00 Noon	175	280	430	50	55	10
6-6	10:30 A.M.	140	190	390	35	50	30
6-7	1:00 P.M.	135	185	525	45	40	115
6-8	12:00 Noon	100	130	420	40	30	75
6-9	2:00 P.M.	100	125	430	35	10	70
6-10	11:30 A.M.	70	80	270	10	10	15
6-11	-	-	-	-	-	-	-
6-12	-	-	-	-	-	-	-
6-13	9:30 A.M.	170	180	580	80	30	50
14-day Total		7,140	-	4,840	210	610	205

Run VI. - continued

7	8	9	10	11	12	13	14
0	70	0	225	180	0	0	175
150	150	0	660	580	75	50	380
35	150	0	740	750	310	170	490
160	280	50	700	700	550	50	500
160	250	190	660	740	680	80	530
110	190	150	710	770	600	150	630
90	160	180	580	690	500	100	660
90	170	180	500	720	540	90	800
30	100	130	250	430	380	70	680
30	70	90	110	210	270	20	500
40	90	120	120	170	230	30	400
40	120	170	150	180	300	0	410
60	110	160	100	140	260	50	260
50	150	230	120	155	350	40	240
80	125	210	75	90	240	20	150
70	120	270	80	100	280	20	135
40	110	190	20	50	160	0	80
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
210	450	700	110	170	370	20	200
14-day Total							
1,045	2,050	1,650	5,625	6,415	5,045	900	6,655

Run VII. June 22, 1956, 6:00 P.M.

Date	Time	Gas Production of Samples Indicated (Milliliters measured at about 25° C. and 74 cm. Mercury, including water vapor)				
		1	2	3	4	5
6-23	12:30 P.M.	380	90	0	0	0
6-24	12:30 P.M.	650	330	5	5	5
6-25	1:30 P.M.	670	410	0	35	55
6-26	12:30 P.M.	600	430	60	0	0
6-27	12:30 P.M.	560	425	55	0	0
6-28	1:00 P.M.	450	380	80	0	0
6-29	1:00 P.M.	335	350	105	0	0
6-30	1:00 P.M.	200	350	100	0	0
7-1	2:30 P.M.	140	275	80	0	0
7-2	12:00 Noon	75	200	70	20	0
7-3	12:30 P.M.	130	90	80	25	0
7-4	12:30 P.M.	85	125	110	45	0
7-5	3:00 P.M.	50	80	105	50	0
7-6	12:00 Noon	60	90	90	35	0
7-7	12:30 P.M.	110	135	130	25	25
7-8	-	-	-	-	-	-
7-9	11:00 A.M.	240	205	175	30	35
14-day Total		4,385	3,625	940	215	60

Run VII - continued

8	9	10	11	12	13
275	85	40	170	0	370
300	60	60	660	5	570
260	40	20	660	0	480
200	30	0	600	0	450
150	10	10	575	0	340
130	15	5	480	5	250
100	30	0	330	0	160
70	10	0	210	0	110
50	5	5	130	10	80
30	0	0	90	20	50
35	10	0	80	30	70
50	15	0	100	35	70
35	5	5	70	25	40
25	0	0	35	15	30
35	10	0	55	25	55
-	-	-	-	-	-
60	10	0	-	35	-
14-day Total					
1,710	315	145	4,190	145	3,070

Note: Samples 6, 7, and 14 gave no significant gas production.

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